

S PN=JP 5097644  
S2 1 PN=JP 5097644  
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DIALOG(R) File 351:Derwent WPI  
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009470793  
WPI Acc No: 1993-164332/ 199320  
UV blocking cosmetic compsn. with no greasiness or stickiness - contains  
alkyl modified polycarboxyvinyl polymer, water insol. powder, oil, water,  
oil-soluble UV absorbent

Patent Assignee: KAO CORP (KAOS )  
Number of Countries: 001 Number of Patents: 001  
Patent Family:

Patent No	Kind	Date	Applicat No	Kind	Date	Week
JP 5097644	A	19930420	JP 91263729	A	19911011	199320 B

Priority Applications (No Type Date): JP 91263729 A 19911011

Patent Details:  
Patent No Kind Lan Pg Main IPC Filing Notes  
JP 5097644 A 4 A61K-007/42

Abstract (Basic): JP 5097644 A

Compsn. comprises: alkyl modified carboxyvinylpolymer, powder  
insoluble in water and oil, oil soluble UV ray absorbing agent and  
water.

USE - UV prevention effect is kept for long time without causing  
stickiness or greasiness

Dwg.0/0

Derwent Class: A96; D21

International Patent Class (Main): A61K-007/42

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**CLAIMS**

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[Claim(s)]

[Claim 1] Following component (A) - (D)

- (A) The charge of ultraviolet-rays defense makeup containing alkyl denaturation carboxyvinyl polymer  
(B) water and (fine-particles C) oil solubility ultraviolet ray absorbent (D) water insoluble to oil.
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**DETAILED DESCRIPTION**

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the charge of ultraviolet-rays defense makeup which is sticky to the skin, and jarring and \*\*\*\* to apply excel [ charge ] in a feeling of use few, and the ultraviolet-rays defense effectiveness maintains.

[0002]

[Description of the Prior Art] In recent years, the various charges of ultraviolet-rays defense makeup are developed, and it is used in order to prevent this, since suntan causes silverfish and buckwheat dregs. In this, the durability of the ultraviolet-rays defense effectiveness of the charge of emulsification makeup of a water-in-oil type is high. However, a feeling of use was not enough and there was a fault that stickiness arose after desiccation, especially at the dryness time. On the other hand, although the charge of oil-in-water type emulsification makeup excelled [ stickiness ] in the feel side few, the durability of the ultraviolet-rays defense effectiveness was not what can not necessarily be satisfied.

[0003]

[Problem(s) to be Solved by the Invention] Therefore, the purpose of this invention is to find out the charge of ultraviolet-rays defense makeup having the durability of a good feeling of use, and the ultraviolet-rays defense effectiveness.

[0004]

[Means for Solving the Problem] The charge of makeup which contains water in fine particles insoluble to an alkyl denaturation carboxyvinyl polymer, water, and oil and an ultraviolet ray absorbent list found out having a good feeling of use, and the outstanding durability of the ultraviolet-rays defense effectiveness, and this invention persons completed this invention, as a result of inquiring wholeheartedly in view of the above-mentioned actual condition.

[0005] That is, this invention is following component (A) - (D).

(A) Offer the charge of ultraviolet-rays defense makeup containing alkyl denaturation carboxyvinyl polymer (B) water and (fine-particles C) oil solubility ultraviolet ray absorbent (D) water insoluble to oil.

[0006] (A) component slack alkyl denaturation carboxyvinyl polymer used by this invention is the thickener of a drainage system, and also has the capacity which distributes an oil by the drainage system. Specifically, the polymerization product (JP,59-232107,A) guided from a, following b, and following c is mentioned as a desirable thing.

The ester c of a, the olefin nature unsaturated-carboxylic-acid monomer b, an acrylic acid or a methacrylic acid, and the alcohol of carbon numbers 10-30, an olefin nature polyfunctional monomer (cross linking agent)

As for this a, b, and c, it is still more desirable to carry out a polymerization as a= 95.9 - 98.8 % of the weight (for "%" to only show hereafter), b= 1 - 3.5%, and c= 0.1 - 0.6%. In addition, as a commercial thing, Carbopol 1342, PENYUREN TRI, and PENYUREN TRII (all good rich company make) are mentioned.

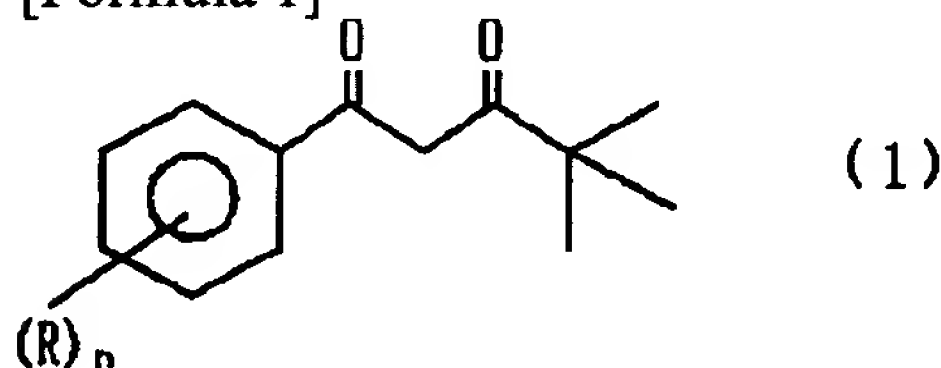
[0007] (A) Two or more sorts may be mixed and used for at least one sort of alkyl denaturation carboxyvinyl polymers of a component, and considering as 0.01 - 5% is desirable, and in order to raise a feeling of use, and durability further, considering as 0.1 - 1.0% is desirable [ loadings ]. If out of range, the feeling durability of use is not [ 0.01 - 5% of ] sometimes enough.

[0008] (B) Especially if fine particles insoluble to the water and the oil of a component are insoluble matter substantially, they will not be restricted to water and oils, such as a pigment and an ultraviolet ray absorbent. For example, titanium oxide, ferrous oxide, ultramarine blue, a zinc white, magnesium oxide, a zirconium dioxide, Inorganic pigments, such as a mica, a sericite, talc, a silica, a kaolin, chromium hydroxide, and carbon black, Ultraviolet ray absorbents, such as particle titanium oxide, a particle zinc oxide, and a thin film integrated circuit zinc oxide, Organic fine particles, an organic pigment, etc. of nylon powder, polymethylmethacrylate, a styrene-divinylbenzene copolymer, polyethylene powder, and the poly methyl silsesquioxane powder (for example, the Toshiba Silicone make, a toss pearl, etc.) are mentioned. In addition, these fine particles may carry out hydrophobing processing using the suitable hydrophobic matter. (B) As for the loadings of the fine particles of a component, considering as 1 - 10% is desirable, and, as for particle size, it is still more desirable to be referred to as 0.01-10 micrometers.

[0009] (C) As an oil solubility ultraviolet ray absorbent of a component For example, p-aminobenzoic-acid ethyl, PARAJI methylamino benzoic-acid octyl [Escarol (ESCALOL) 507 (VANDYK)], Cinoxate, PARAMETOKISHI cinnamic acid octyl [Escarol 557 and Parsol (Parsol) MCX (GIVAUDAN)], 2-(2-hydroxy-5-methylphenyl) benzotriazol, Oxybenzone [Escarol 567 and spectra SORUBU (Spectra-Solb) UV 9 (American Cyanamid)], urocanic acid, urocanic acid ethyl, a benzophenone, and a tetra-hydroxy benzophenone -- [ -- for example The next general formula given in Uvinul D50(BASF A.G.)], 4-t-butyl-4'-methoxy benzoyl methane [Parsol 1789], JP,2-212579,A, and JP,3-188041,A (1)

[0010]

[Formula 1]



[0011]

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**TECHNICAL FIELD**

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[Industrial Application] This invention relates to the charge of ultraviolet-rays defense makeup which is sticky to the skin, and jarring and \*\*\*\* to apply excel [ charge ] in a feeling of use few, and the ultraviolet-rays defense effectiveness maintains.

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**EFFECT OF THE INVENTION**

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[Effect of the Invention] The charge of ultraviolet-rays defense makeup of this invention is an outstanding charge of makeup which is sticky, and is excellent in a feeling of use -- there are little jarring and \*\*\*\* to apply -- to the skin, and the ultraviolet-rays defense effectiveness maintains.

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**TECHNICAL PROBLEM**

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**MEANS**

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[Means for Solving the Problem] The charge of makeup which contains water in fine particles insoluble to an alkyl denaturation carboxyvinyl polymer, water, and oil and an ultraviolet ray absorbent list found out having a good feeling of use, and the outstanding durability of the ultraviolet-rays defense effectiveness, and this invention persons completed this invention, as a result of inquiring wholeheartedly in view of the above-mentioned actual condition.

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**EXAMPLE**

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[Example]

In example of reference 11-(3, 4-dimethoxy phenyl)-4, and 4-dimethyl pentane -1, and a 3-dione [general formula (1)  $R=OCH_3$ , synthetic : stirring equipment of thing] of  $n=2$ , With the 200ml \*\* 3 TSU opening flask equipped with a dropping funnel, a reflux condenser, and nitrogen installation tubing Stirring mixing of the 60% sodium hydride [ 2.45 ] (61mmol) and 3, 10g [ of 4-dimethoxy methyl benzoates ] (51mmol), and anhydrous tetrahydrofuran 100ml was carried out under the nitrogen air current, and bottom pinacolone of heating reflux 6.1g (61mmol) was dropped. After cooling radiationally after 7-hour heating reflux and adding 30ml of 2N-hydrochloric acids, chloroform extracted twice. The solvent was distilled off for the extract after desiccation with anhydrous sodium sulfate, and the rough product was obtained. The hexane was added to this, it recrystallized by having condensed filtrate after filtering insoluble matter, and 8.9g of colorless needle crystal of the purpose compound was obtained (65% of yield).

Melting point : 52.3-53.3-degree-CIR : 1600, 1520, 1470, 1450, 1370, 1300, 1270, 1220 and 1190, 1130, 890, 790, 7301 H-NMR( $CDCl_3$ ,  $\delta$ ): 1.26 (9H, s, t- $C_4H_9$ ), (nuKBr,  $cm^{-1}$ ) 3.95 (3H, s,  $OCH_3$ ) 3.96 (3H, s,  $OCH_3$ ), 6.24 (1H, s), 6.90 (1H, d,  $J=8.4Hz$ ), 7.49 (1H, s), 7.51 (1H, d,  $J=8.4Hz$ ). elemental-analysis calculated value (%) C; 68.16, H; 7.63 actual measurement (%) C; 68.23, H; 7.60 [0018] The charge of ultraviolet-rays defense makeup of the presentation shown in an example 1 - the 3 following table 1 is manufactured according to the following manufacturing method, and the result of having evaluated a feeling of use by ten panelists is shown in Table 1.

[0019]

[Table 1]

(%)

組 成	実施例 1	実施例 2	実施例 3	比較例 1	比較例 2
(1) アルキル変性カルボキシビニルポリマー (カーボボール1342) カルボキシビニルポリマー (カーボボール941)	0.4 —	0.4 —	0.4 —	0.4 —	— 0.4
(2) タルク 酸化チタン トスパール (シリコーン樹脂微粒子)	5.0 — —	— 5.0 —	— — 5.0	— — —	— — —
(3) メトキシケイ皮酸オクチル* <sup>1</sup> ジメチルアミノ安息香酸オクチル* <sup>2</sup> 参考例 1 の化合物	3.0 2.0 5.0	3.0 2.0 5.0	3.0 2.0 5.0	3.0 2.0 5.0	3.0 2.0 5.0
(4) 精製水	バランス	バランス	バランス	バランス	バランス
(5) KDH	0.15	0.15	0.15	0.15	0.15
べたつかない ぬるつかない 持続性	○ ○ ○	△ ○ ○	○ ○ ○	× × △	—* <sup>3</sup> —* <sup>3</sup> —* <sup>3</sup>

\*1: パーソールMCX

\*2: エスカロール507

\*3: 均一に乳化・分散しないため評価不能

[0020] Valuation-basis [ of a feeling of use ] O: Good (seven or more persons answered it as good)

\*\*: It is good (4-6 persons answered it as good) a little.

x: It is inferior. (below trinominal answered it as good)

An organic solvent extracts, 7 hours after carrying out constant-rate spreading at a lasting valuation-basis frame. It is a quantum about the amount of UV absorbents at HPLC.

O: -- a survival rate -- more than 80%\*\*: -- a survival rate -- less than [ 80% 50% or more ] x: -- a survival rate -- less than 50% manufacturing method: -- heat 1. (3), make it liquefied, and make this distribute (2)

(1) is made to dissolve or distribute 2. (4) at 50-60 degrees C.

1. is gradually added and stirred to 3.2., and it neutralizes by (5).

[Translation done.]

(19) 日本国特許庁 ( J P )

(12) 公 開 特 許 公 報 ( A )

(11) 特許出願公開番号

特開平5-97644

(43) 公開日 平成 5 年 (1993) 4 月20日

(51) Int.Cl. <sup>5</sup>	識別記号	庁内整理番号	F I	技術表示箇所
A 6 1 K 7/42		7252-4C		

審査請求 未請求 請求項の数 1 (全 4 頁)

(21) 出願番号	特願平3-263729	(71) 出願人	000000918 花王株式会社 東京都中央区日本橋茅場町 1 丁目14番10号
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(54) 【発明の名称】 紫外線防御化粧品

(57) 【要約】

【構成】 次の成分 (A) ~ (D)

(A) アルキル変性カルボキシビニルポリマー

(B) 水及び油に不溶である粉体

(C) 油溶性紫外線吸収剤

(D) 水

を含有する紫外線防御化粧品。

【効果】 本発明の紫外線防御化粧品は、皮膚に対して、べたつき、きしみ、ぬるつきが少ない等使用感に優れ、かつ紫外線防御効果が持続する優れた化粧品である。

1

【特許請求の範囲】

【請求項1】 次の成分(A)～(D)

(A) アルキル変性カルボキシビニルポリマー

(B) 水及び油に不溶である粉体

(C) 油溶性紫外線吸収剤

(D) 水

を含有する紫外線防御化粧料。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、皮膚に対してべたつき、きしみ、ぬるつきが少なく使用感に優れ、かつ紫外線防御効果が持続する紫外線防御化粧料に関する。

【0002】

【従来の技術】近年においては、日焼けはシミ、ソバカスの原因となることから、これを防止するため、種々の紫外線防御化粧料が開発され、用いられている。この中では、油中水型の乳化化粧料は紫外線防御効果の持続性は高い。しかしながら、使用感が十分でなく、特に乾き際、乾燥後にべたつきが生じるという欠点があった。一方、水中油型乳化化粧料はべたつき等が少なく感触面で優れているが、紫外線防御効果の持続性は必ずしも満足できるものではなかった。

【0003】

【発明が解決しようとする課題】従って本発明の目的は、良好な使用感と紫外線防御効果の持続性を併せ持つ紫外線防御化粧料を見出すことにある。

【0004】

【課題を解決するための手段】本発明者らは上記実状に鑑み鋭意研究を行なった結果、アルキル変性カルボキシビニルポリマー、水及び油に不溶な粉体、紫外線吸収剤並びに水を含有する化粧料が、良好な使用感と優れた紫外線防御効果の持続性を合せ持つことを見出し本発明を完成した。

【0005】すなわち本発明は、次の成分(A)～(D)

(A) アルキル変性カルボキシビニルポリマー

(B) 水及び油に不溶である粉体

(C) 油溶性紫外線吸収剤

(D) 水

を含有する紫外線防御化粧料を提供するものである。

【0006】本発明で用いる(A)成分たるアルキル変性カルボキシビニルポリマーは、水系の増粘剤であり、水系で油を分散する能力をも有するものである。具体的には、例えば次のa、b及びcから誘導される重合生成物(特開昭59-232107号公報)が好ましいものとして挙げられる。

a、オレフィン性不飽和カルボン酸モノマー

b、アクリル酸又はメタクリル酸と炭素数10～30のアルコールとのエステル

c、オレフィン性多官能性モノマー(架橋剤)

2

このa、b及びcは、 $a=95.9\sim 98.8$ 重量%(以下、単に「%」で示す)、 $b=1\sim 3.5\%$ 、 $c=0.1\sim 0.6\%$ として重合することが更に好ましい。なお、市販のものとしては、カーボボール1342、ペニュレンTRI、ペニュレンTRII(いずれもグッドリッチ社製)が挙げられる。

【0007】(A)成分のアルキル変性カルボキシビニルポリマーは、1種でも2種以上を混合して用いても良く、配合量は $0.01\sim 5\%$ とすることが好ましく、更に使用感、持続性を向上させるためには $0.1\sim 1.0\%$ とすることが好ましい。 $0.01\sim 5\%$ の範囲外では使用感持続性が十分でないことがある。

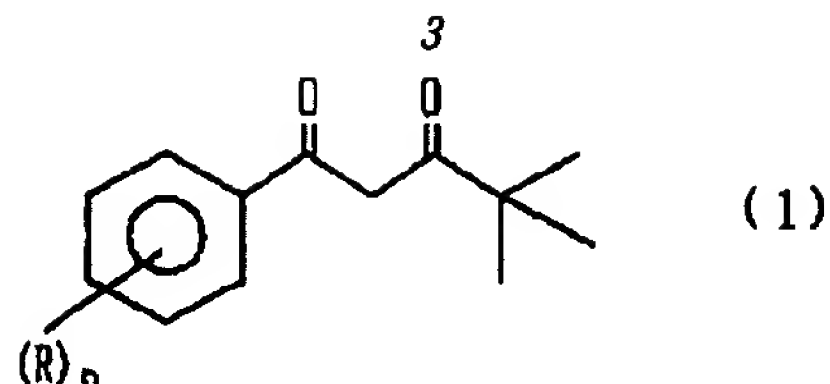
【0008】(B)成分の水及び油に不溶である粉体は、顔料、紫外線吸収剤等の水及び油に実質的に不溶な物質であれば特に制限されず、例えば酸化チタン、酸化鉄、群青、亜鉛華、酸化マグネシウム、酸化ジルコニウム、マイカ、セリサイト、タルク、シリカ、カオリン、水酸化クロム、カーボンブラック等の無機顔料、微粒子酸化チタン、微粒子酸化亜鉛、薄片状酸化亜鉛等の紫外線吸収剤、ナイロンパウダー、ポリメチルメタクリレート、スチレンービニルベンゼン共重合体、ポリエチレン粉末、ポリメチルシルセスキオキサン粉末(例えば、東芝シリコーン社製、トスパール等)の有機粉体及び有機顔料等が挙げられる。なお、これらの粉体は適当な疎水性物質を用い疎水化処理したものであっても良い。

(B)成分の粉体の配合量は $1\sim 10\%$ とすることが好ましく、更に粒径は $0.01\sim 10\mu\text{m}$ とすることが好ましい。

【0009】(C)成分の油溶性の紫外線吸収剤としては、例えば、パラアミノ安息香酸エチル、パラジメチルアミノ安息香酸オクチル〔エスカロール(ESCALOL)507(VANDYK社)〕、シノキサート、パラメトキシ桂皮酸オクチル〔エスカロール557、パーソール(Parsol)MCX(GIVAUDAN社)〕、2-(2-ヒドロキシ-5-メチルフェニル)ベンゾトリアゾール、オキシベンゾン〔エスカロール567、スペクトラソルブ(Spectra-Solb)UV9(American Cyanamid社)〕、ウロカニン酸、ウロカニン酸エチル、ベンゾフェノン、テトラヒドロキシベンゾフェノン〔例えば、ユビナールD50(BASF社)〕、4-tert-ブチル-4'-メトキシベンゾイルメタン〔パーソール1789〕、特開平2-212579号公報、特開平3-188041号公報に記載の次の一般式(1)

【0010】

【化1】



【0011】〔式中、Rは水酸基、炭素数1～8のアルコキシ基、炭素数1～8のアルケニルオキシ基若しくは（ポリオキシアルキレン）オキシ基を示し、又は2個のRで $\alpha$ -メチレンジオキシ基を形成してもよく、nは1～3の整数を示す〕で表わされるベンゾイルピナコロン誘導体等が挙げられる。

【0012】これら、紫外線吸収剤は1種のみ用いても良いが、異なる性質の2種以上を組み合わせ用いる方が、夫々の特性を生かせるため有利である。配合量は1～30%とすることが好ましく、特に5～30%とすることが好ましい。この量が1%未満であると紫外線防御効果が十分でなく、30%を超えて配合しても効果の向上は少なく好ましくない。

【0013】(D)成分たる水は40～90%配合することが好ましく、特に70～85%配合することが好ましい。

【0014】本発明の紫外線防御化粧料には、上記必須成分の他本発明の効果を妨げない限り、種々の油成分、香料、防腐剤、保湿剤、乳化安定剤、薬効成分、着色剤、pH調整剤等を適宜選択して配合することもできる。

【0015】本発明の紫外線防御化粧料は、常法により加熱、分散、混合等の操作を組み合わせ製造することができる。

【0016】

【発明の効果】本発明の紫外線防御化粧料は、皮膚に対して、べたつき、きしみ、ぬるつきが少ない等使用感に優れ、かつ紫外線防御効果が持続する優れた化粧料であ

る。

【0017】

【実施例】

参考例1

1-(3,4-ジメトキシフェニル)-4,4-ジメチルペンタン-1,3-ジオン〔一般式(1)においてR=OCH<sub>3</sub>, n=2のもの〕の合成：攪拌装置、滴下ロート、還流冷却器及び窒素導入管を備えた200ml容三口フラスコにて、60%水素化ナトリウム2.45g(61mmol)、3,4-ジメトキシ安息香酸メチル10g(51mmol)及び無水テトラヒドロフラン100mlを窒素気流下、攪拌混合し、加熱還流下ピナコロン6.1g(61mmol)を滴下した。7時間加熱還流後放冷し、2N-塩酸30mlを加えた後、クロロホルムで2回抽出した。抽出液を無水硫酸ナトリウムで乾燥後、溶媒を留去し、粗生成物を得た。これにヘキサンを加え、不溶物を濾過後、濾液を濃縮し再結晶を行い、目的化合物の無色針状結晶8.9gを得た(収率65%)。

融点：52.3～53.3℃

IR( $\nu_{\text{KBr}}$ , cm<sup>-1</sup>): 1600, 1520, 1470, 1450, 1370, 1300, 1270, 1220, 1190, 1130, 890, 790, 730

<sup>1</sup>H-NMR(CDCl<sub>3</sub>,  $\delta$ ): 1.26(9H, s, t-C<sub>4</sub>H<sub>9</sub>), 3.95(3H, s, OC<sub>4</sub>H<sub>9</sub>), 3.96(3H, s, OCH<sub>3</sub>), 6.24(1H, s), 6.90(1H, d, J=8.4Hz), 7.49(1H, s), 7.51(1H, d, J=8.4Hz).

元素分析

計算値(%) C;68.16, H;7.63

実測値(%) C;68.23, H;7.60

【0018】実施例1～3

下記表1に示す組成の紫外線防御化粧料を下記製造法により製造し、使用感をパネラー10名により評価した結果を表1に示す。

【0019】

【表1】

(%)

組 成	実施例 1	実施例 2	実施例 3	比較例 1	比較例 2
(1) アルキル変性カルボキシビニルポリマー (カーボポール1342) カルボキシビニルポリマー (カーボポール941)	0.4 —	0.4 —	0.4 —	0.4 —	— 0.4
(2) タルク 酸化チタン トスパール (シリコーン樹脂微粒子)	5.0 — —	— 5.0 —	— — 5.0	— — —	— — —
(3) メトキシケイ皮酸オクチル* <sup>1</sup> ジメチルアミノ安息香酸オクチル* <sup>2</sup> 参考例 1 の化合物	3.0 2.0 5.0	3.0 2.0 5.0	3.0 2.0 5.0	3.0 2.0 5.0	3.0 2.0 5.0
(4) 精製水	バランス	バランス	バランス	バランス	バランス
(5) KDH	0.15	0.15	0.15	0.15	0.15
べたつかない	○	△	○	×	—* <sup>3</sup>
ぬるつかない	○	○	○	×	—* <sup>3</sup>
持続性	○	○	○	△	—* <sup>3</sup>

\*<sup>1</sup>: パーソールMCX\*<sup>2</sup>: エスカロール507\*<sup>3</sup>: 均一に乳化・分散しないため評価不能

## 【0020】使用感の評価基準

○: 良 (7名以上が良と答えた)

△: やや良 (4~6名が良と答えた)

×: 劣る (3名以下が良と答えた)

## 持続性の評価基準

額に一定量塗布して7時間後に有機溶媒で抽出。HPLCでUV吸収量を定量。

○: 残存率が80%以上

△: 残存率が50%以上80%未満

×: 残存率が50%未満

## 製造法:

1. (3) を加熱し液状にし、これに(2)を分散させる。

30 2. (4) を(1)に50~60℃にて溶解又は分散させる。

3. 2. に1. を徐々に添加、攪拌し、(5)で中和する。

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